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RECONDITIONING AND REUSE OF FOOD PROCESSING BRINES

Walter A. Mercer

National Canners Association
Berkeley, California

May 1969

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May 1969
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16. Abstract

Storage brines and processing waters from the production of canned and glass packed olives were treated with activated carbon. The reuse potential of reconditioned brines was evaluated. Reconditioned concentrated storage brines can be used to store freshly harvested olives for several months. Reconditioned brines of lower salt content were reused with no detectable effect on the quality of the final product.

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PREFACE

The advice, counsel and cooperation of the following members of the Technical Advisory Committee of the California Olive Association is gratefully acknowledged:

Orrin Scott	Bell Packing Company
Noel Graves	California Canners and Growers
J. R. Webster	Consolidated Olive Growers
Ralph Fusano	Cristo Fusano & Sons
William Perry	Early California Foods, Inc.
James Chung	Libby, McNeill & Libby
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Reference to a company or product name does not imply approval or recommendation of the product by the U.S. Department of the Interior or the U. S. Department of Agriculture and does not imply that there are no other suitable products available commercially.

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INTRODUCTION

Substantial quantities of sodium salts (chloride, hydroxide) are used in many food processing operations. The disposal of the saline liquid wastes from these operations, without causing water pollution, is a problem of increasing complexity. The primary difficulty in the disposal of saline liquid wastes is the non-biodegradable character of sodium chloride and sodium hydroxide. Incorporation of sodium salts into cells of microorganisms during biological stabilization serves only to transpose the saline pollution potential from a liquid waste to a solid waste. The only economical "solution" to the disposal of saline liquid waste is the discharge to salt water receiving bodies or dilution with large volumes of fresh water. Many food processors at inland locations have inadequate fresh water dilution capacity available to assimilate discharges of saline liquid wastes from their operations. These food processors must change their production methods to reduce the discharge of saline wastes, move to sites near salt water, or go out of business.

The work reported here is confined to treatment of brines produced during the production of canned olives. This industry was selected for the initial study due to the urgent need for ways to alleviate the critical pollution potential of saline liquid wastes generated in the Central Valley of California. Substantial quantities of salt are also used in the production of pickles, sauerkraut and other foods in many areas of the United States. Information developed in this demonstration grant should be useful in the solution of potential water pollution problems of all sectors of the food processing industry which use brines in direct contact with food materials.

Increased total saline loads from population growth and changes in agricultural practices, as well as more stringent discharge requirements, are responsible for the critical disposal problem in the Central Valley of California. Long range research is now underway (at the National Canners Association and at the University of California, Davis) to provide storage systems for olives which do not contain appreciable quantities of sodium chloride. These systems (acidified water containing bacteriostatic organic compounds; chemical fertilizer salts; or controlled atmosphere and temperature storage) will produce waste streams which can be disposed of more readily than sodium chloride brines. This research is in an exploratory stage and, even if successful, will require five to ten years to become the general commerical

practice of the olive canning industry. Until these methods are fully developed and tested on a commercial scale, it will be necessary to reuse or to dispose of strong saline brines in a way which satisfies regulatory agencies.

The existing technology for handling and disposal of food processing brines was reviewed and evaluated. The most promising method for immediate relief of saline pollution problems was based on exploratory research done at the Western Utilization Research and Development Division, U. S. Department of Agriculture. Small volumes of olive storage brines were passed through a column containing activated carbon. The purified brine was used by a commercial canner to successfully store freshly harvested olives. The final canned product from olives stored in the reconditioned brine was of acceptable quality. The high-lights of this exploratory study have been published (1).

Further developmental work on the use of activated carbon for reconditioning olive brines was reported from the USDA Laboratory (2); these studies were conducted to develop information needed to design a pilot plant scale unit capable of processing 5,000 gallons of brine per week. Results of further studies on carbon treatment of olive brines by the USDA Western Regional Laboratory were reported in 1968 (3).

The economics of reconditioning olive brines by activated carbon treatment appear attractive based on figures derived from the small scale studies. A larger scale operation was necessary to demonstrate technical feasibility under commercial conditions and to develop more accurate cost factors.

The composition of storage brines and processing waters is different for each olive canning operation. To obtain representative operational experience and cost figures, the brine reconditioning unit will be installed at four different locations during the demonstration grant period.

DESIGN AND CONSTRUCTION OF DEMONSTRATION SCALE BRINE RECONDITIONING UNIT

The original design for a brine reconditioning unit (having as its main component a carbon column section 20 feet long and 16 inches in diameter) was made by the Engineering and Development Laboratory, WURDD, ARS, USDA in Albany, California. This drawing was part of the proposal submitted to the Federal Water Pollution Control Administration in April, 1967.

At the time of the acceptance of the FWPCA demonstration grant offer by NCA in February, 1968, it was found that severe corrosion occurred in stainless steel equipment exposed to mixtures of carbon and salt brine. It was necessary to convert the original drawing which had specified stainless steel as the material of construction to drawings based on fiber glass reinforced food grade resin. Fortunately, a plastic fabricator provided a set of drawings which served to obtain bids from potential suppliers of the equipment. After FWPCA review of specifications, requests for bids were sent to three companies in May. The quotations received by June 1 were summarized for FWPCA review. On June 12, FWPCA approval of the California Blowpipe and Steel Company, Inc. of Escalon, California as the general contractor was received. The sub-contractor for the fabrication of the plastic components was the Heil Process Equipment Corporation of Cleveland, Ohio.

Working drawings for the plastic components and the support structure were reviewed and modifications suggested during July and August. Anticipating an August 15 delivery date, all necessary accessory components (air compressor, delivery pump, piping, guy cable and turnbuckles, compressed air reduction manifold and control panel, activated carbon, plastic floats screen and filter cloth) were purchased and delivered before August 1 to Lindsay, California where the unit was to be initially located. After a series of delays in fabrication and shipping of the plastic components, the unit was erected on September 30, 1968.

INSTALLATION OF THE BRINE RECONDITIONING UNIT

The unit was used first at Lindsay, California during the period September 30 to December 30, 1968. After the major components had been mounted in the support structure and the unit set up and secured by cables, the accessory components were installed. A 1500 gallon capacity plastic swimming pool served as an influent settling tank. Brine was removed from the settling tank (through a stainless steel cloth filter) with a Monyo pump (Model Number 1L 3 SSQ) having a delivery rate of 0.5 to 3.5 gallons per minute. The brine was delivered to the bottom of the carbon column through a hose line and distributed with a circular, perforated stainless steel pipe.

Compressed air was supplied by a one horsepower Ingersoll Rand air compressor (Model 1 HPC1) at 50 pounds pressure. A control panel having a manifold of pressure regulators and valves was used to control air pressure to the blow pot and to the air activated valves. Copper tubing was used to deliver compressed air to the Keystone valves. Water slurries of activated carbon (Pittsburgh Type CAL 12 x 40) were prepared in a plastic tank and delivered to the blow pot. Compressed air was used to force the slurry to the hopper on the top of the column through a 1 inch diameter polyvinyl chloride pipe. The overflow return from the hopper to the slurry tank was transported through a 3 inch polyvinyl chloride pipe. The carbon was held in the column by polyethylene beads (Float-Screen) and stainless steel cloth. The effluent brine was delivered to collecting tanks through a one inch hose. Sampling ports on the side of the main column (number 1 for the lowest and 5 for the highest) were used to collect samples representative of brine at that level in the carbon bed.

Figures 1 and through 3 show the final assembled unit as it appeared on location in Lindsay. Figure 1 shows an overall view of the unit and accessory equipment. Figure 2 was taken at ground level and shows the air compressor, delivery pump and influent brine tank in the left background. Figure 3 is of the carbon slurry tank and pressure blow pot.

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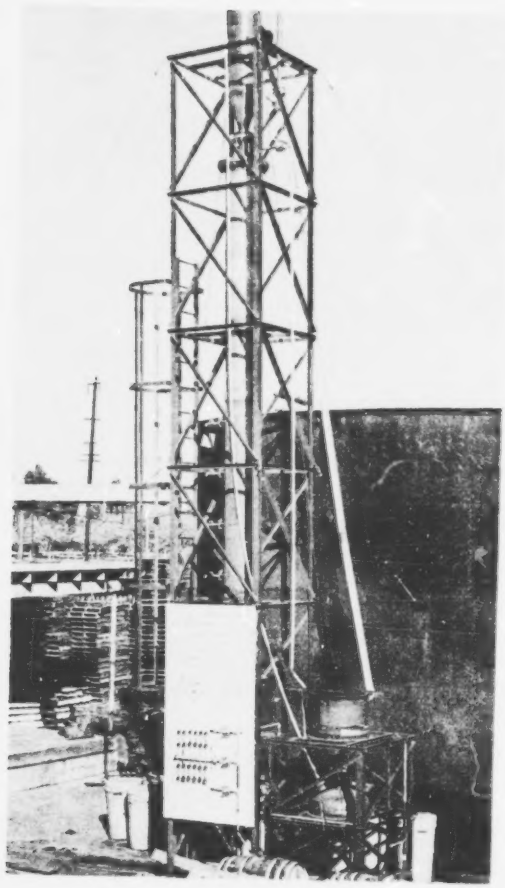


Figure 1. Overall View of Brine Reconditioning Unit

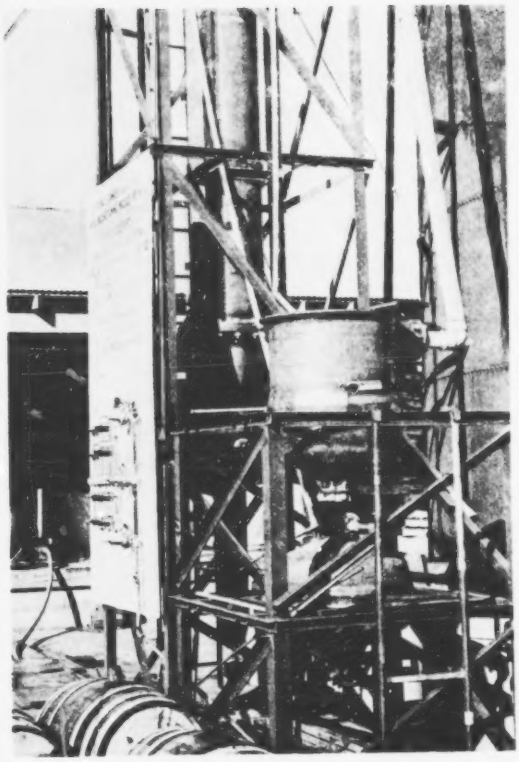
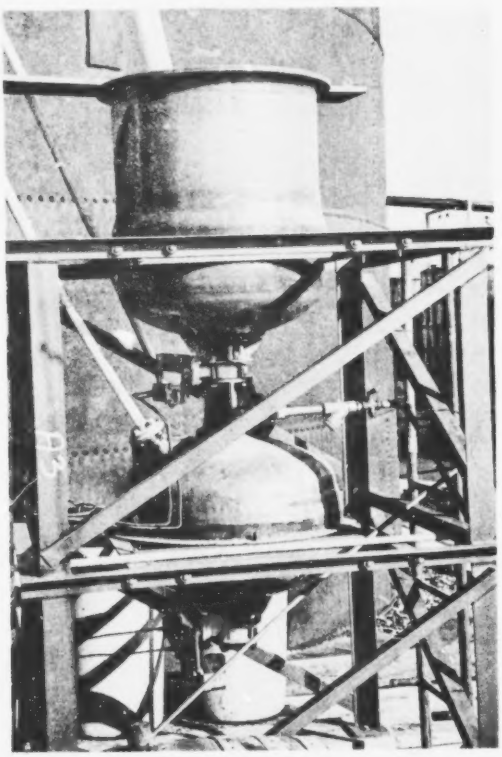


Figure 2.
Lower Portion of Brine
Reconditioning Unit With
Accessory Components
in Background

Figure 3. Carbon Slurry Tank and Blow Pot



SAMPLING AND ANALYSIS PROGRAM

The major objective of this project was the preparation on a commercial scale of reconditioned olive storage and processing brines and evaluation of their reuse potential. The treatment of food processing brines with activated carbon was expected to remove part of the organic compounds present in the brine. The detailed chemical identity of the organic compounds in olive brines has not been established. The actual compounds in olive brines which must be completely or partially removed before the brines can be reused successfully are, as yet, unknown.

It was beyond the scope of this project to isolate, identify and measure individual organic components in used and reconditioned brines. The reuse potential of reconditioned brines was to be established by direct observation of the results of reuse on the quality of the canned olive product. A set of conventional measurements on the brine samples was selected as indicators of gross changes in brine composition after various treatments and re-use applications. The analyses were of the type that could be done readily in a cannery to serve as a quality control tool or as an indication on the suitability of reconditioned brines for reuse.

The following analyses were performed on as many representative samples of brines as time and personnel allowed:

1) chemical oxygen demand; 2) suspended solids; 3) color; 4) pH; 5) total acidity; and 6) salt content.

It was expected that one or a combination of these analyses would correlate well with the reuse potential of reconditioned olive brines. The experimental details for the analyses listed above will be found in the Methodology Section of this report.

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OPERATION OF THE BRINE RECONDITIONING UNIT

Lindsay Storage Brine

The brine reconditioning unit was used first to process high salt content olive storage brine which had been held over from the storage of olives during the 1967 season. The column section of the unit was charged with 1020 pounds of CAL Type 12 x 40 activated carbon and a total of 12,750 gallons of storage brine put through the unit over a period of fourteen days. The flow rate was from 2 to 3 gallons per minute. Six 60-pound portions of fresh carbon were added at intervals during this period after spent carbon had been drained. Samples of influent brine, brine at various sampling ports on the column and effluent brine was collected and analyzed.

The results of analyses on influent and effluent brine samples are tabulated in Tables 1, 2, 3, 4 and shown graphically in Figures 4, 5, 6.

Table 1 lists the subjective color judgments on the brines. The influent brine was dark yellow green in color and the effluent brine ranged from almost water white to a light yellow green. The subjective color observation seem to be a good criteria of brine quality. The objective color measurements are tabulated in Table 2. The color of the reconditioned brine changes during storage and only a few valid readings were obtained before this behavior was noted. More extensive correlations of subjective and objective estimations of brine color is reported for the Visalia storage brines.

The results of the measurement of pH and total acidity of brines are tabulated in Table 3 and shown graphically in Figures 4 and 5. There are sharp increases in pH and decreases in total acidity as brines contact fresh carbon. However, for most of the gallonage delivered the pH and total acidity are similar in both the influent and effluent brines.

The measurement of chemical oxygen demand is tabulated in Table 4 and shown graphically in Figure 6. The chemical oxygen demand of the effluent brine drops sharply after each addition of fresh carbon. The general trend of the chemical oxygen demand is to increase up to the influent level as the gallonage put through the car-

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TABLE 1

COLOR OF RECONDITIONED LINDSAY STORAGE BRINE

Cumulative Gallons Treated	Subjective Color	Cumulative Gallons Treated	Subjective Color
0	Dark Yellow Green	7,175	Cloudy
270	Cloudy	7,200	Cloudy
630	Cloudy	7,775	Light Grey
1,350	Cloudy	8,075	Cloudy
2,010	Cloudy	8,375	Cloudy
2,910	Cloudy	8,400	Cloudy
3,150	Cloudy	8,700	Cloudy
3,270	Light Yellow Green	9,000	Cloudy
3,390	Cloudy	9,150	Light Grey
3,750	Light Yellow Green	9,650	Cloudy
4,110	Cloudy	9,800	Cloudy
4,660	Light Grey	10,985	Light Grey
5,630	Cloudy	11,225	Cloudy
5,930	Cloudy	12,105	Light Yellow Green
6,230	Cloudy	12,753	Light Yellow Green

TABLE 2

LIGHT TRANSMITTANCE FOR OLIVE BRINES

Gallons Treated	Transmittance at 450 mμ
0	76 *
630	75
1, 350	92
3, 150	96
3, 390	92

* 1 part diluted with 5 parts of distilled water

TABLE 3

pH AND TOTAL ACIDITY OF RECONDITIONED LINDSAY STORAGE BRINES

Gallons Treated	pH	% Total Acidity as Lactic Acid
0	4.30	0.39
270	8.00	Not measured
630	6.80	"
1,350	7.35	"
2,010	5.10	"
2,910	4.60	"
3,150	4.60	"
3,270	4.80	"
3,390	6.20	"
3,750	4.50	"
4,110	4.50	0.20
4,660	4.20	0.32
5,630	4.25	0.31
5,930	4.00	0.37
6,230	4.25	0.37
7,175	4.20	0.38
7,200	4.40	0.21
7,775	6.70	0.01
8,075	4.15	0.35
8,375	4.20	0.38
8,400	4.50	0.14
8,700	3.90	0.37
9,000	4.10	0.38
9,150	4.00	0.38
9,650	4.00	0.38
9,800	3.90	0.41
10,985	7.10	0.03
11,225	4.15	0.42
12,105	4.40	0.48
12,753	4.10	0.41

TABLE 4

COD AND SUSPENDED SOLIDS OF RECONDITIONED LINDSAY STORAGE BRINES

Gallons Treated	mg/liter COD	mg/l Suspended Solid
0	18,000	280
270	835	--
630	5,011	--
1,350	--	--
2,010	9,094	220
2,910	10,208	--
3,150	9,002	--
3,270	10,486	--
3,370	5,382	--
3,750	11,162	220
4,110	12,390	--
4,660	10,712	--
5,630	10,400	--
5,930	11,440	--
6,230	10,712	--
7,175	13,104	--
7,200	14,784	164
7,775	6,624	--
8,075	11,136	--
8,375	11,424	--
8,400	7,872	--
8,700	10,848	--
9,000	17,680	196
9,150	13,104	--
9,650	--	--
9,800	--	--
10,985	6,552	--
11,225	11,336	--
12,105	18,944	--
12,753	18,842	--

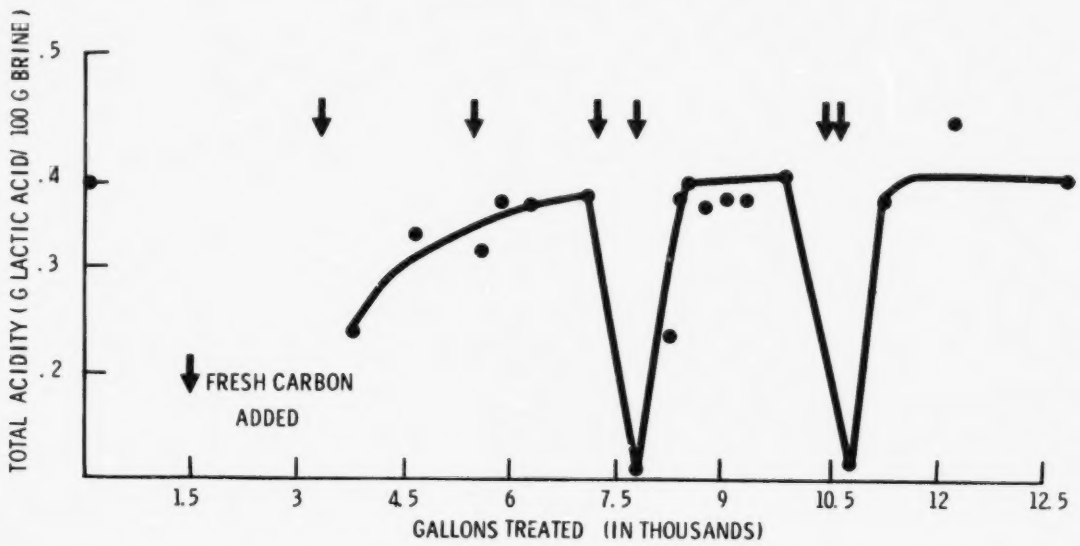

Figure 4. Total Acidity of Reconditioned Lindsay Storage Brine

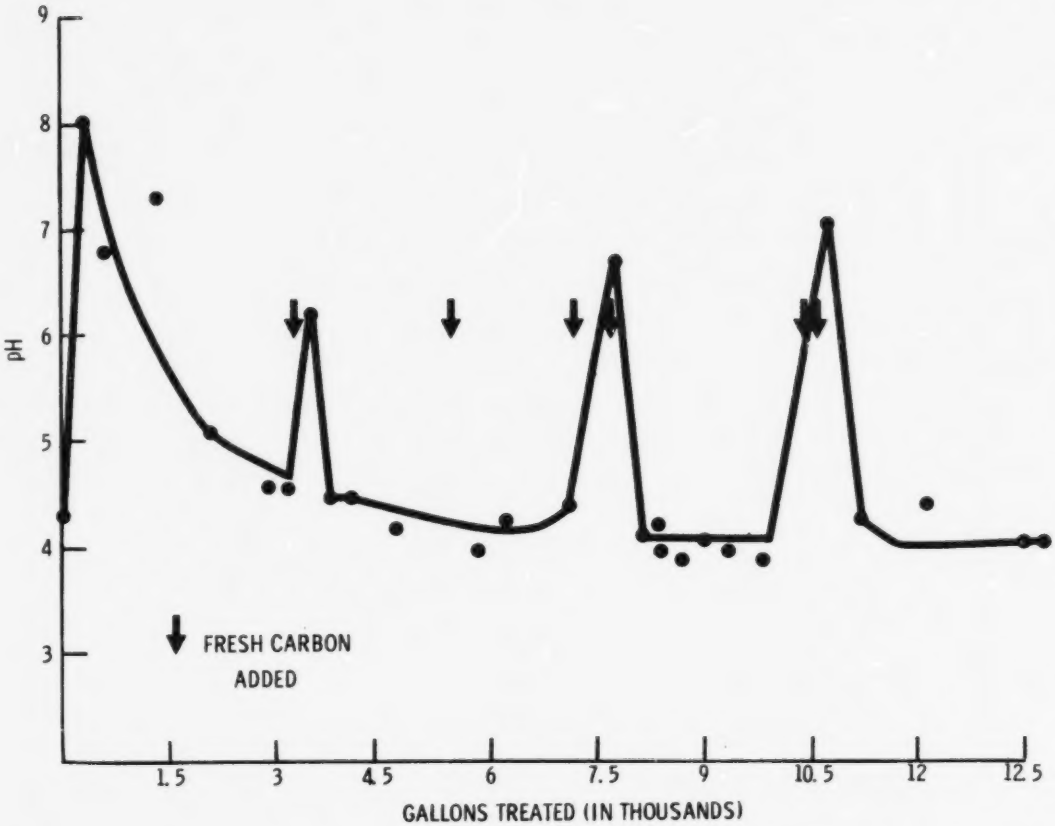

Figure 5. pH of Reconditioned Lindsay Storage Brine

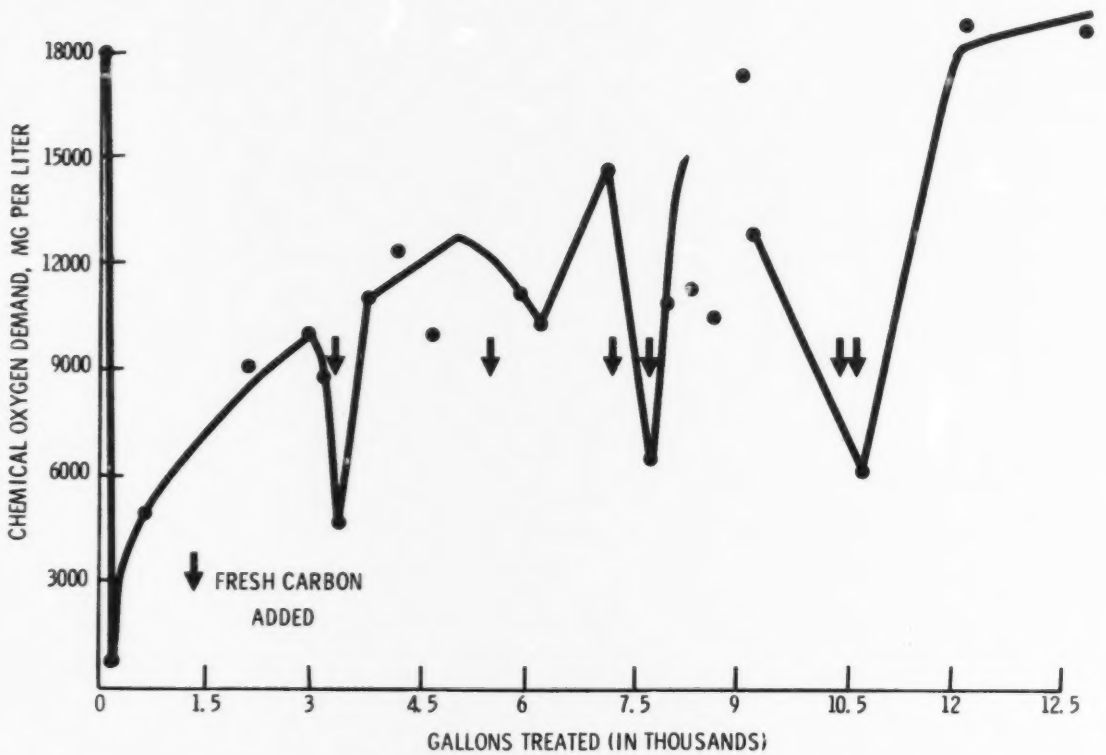

Figure 6. Chemical Oxygen Demand of Reconditioned Lindsay Storage Brine

bon bed increases. The results of these measurements as well as results reported below indicated that considerable brine color is removed without changing the chemical oxygen demand value of the brine.

A few spot analyses for suspended solids are tabulated in Table 4. The value for suspended solids is not markedly changed by passage through the carbon bed. A more extensive series of measurements reported below confirms that there is relatively little change in suspended solids content due to carbon column treatments.

The salt content of storage brines show little change on passage through the carbon bed after a brief initial adsorption period. The influent brine had a range of 5.5 to 6.2 percent sodium chloride and the effluent a range of 5.1 to 6.2 in a series of 85 determinations.

Lindsay Processing Water

The carbon column recycling of Lindsay processing water was performed at Visalia on February 27, 1969 and the reconditioned water returned to Lindsay for evaluation.

Four plastic lined bins, with covers, were filled with spent lye process water and delivered to Visalia. At the plant site, the liquid was siphoned into a 1000 gallon holding tank. One hundred pounds of fresh carbon was placed at the top of the column before pumping of the process water was started. The major part of the carbon within the tower was loaded with caustic and this was eluted as the solution passed through the carbon bed. The processing water was returned in clean bins for a reuse potential evaluation.

A summary of the analytical results are tabulated in Table 5. The dark colored components in the influent are nearly completely adsorbed by the carbon. The pH of the effluent is due to the past history of the carbon in the column which contained high concentrations of sodium hydroxide. Very little salt is contained in the processing water. There was a remarkable reduction (68% average) in COD.

TABLE 5

CARBON COLUMN TREATMENT OF LINDSAY PROCESS WATER

	Light Transmittance at 425 mμ	pH	%NaOH	%Nacl	COD mg/liter	COD Removal
Influent	54.5*	9.3	.022	0.11	6400	----
Effluent Gallons						
60	100	12.0	.334	0.05	835	5568
180	87	11.8	.138	0.10	1949	4457
300	93	11.6	.087	0.12	1949	4454
420	95	11.3	.065	0.12	2320	4083
540	94	11.2	.058	0.11	2877	3526
640	91	11.1	.055	0.12	2320	4083

*Diluted with 9 parts of distilled water

Visalia Storage Brine

The second location of the unit was in Visalia, California during the period December 30, 1968 to April 3, 1969.

A total of 10,000 gallons of olive storage brine was processed with the activated carbon column at Visalia. The flow rate through the carbon bed was maintained at five quarts per minute. The influent brine came from two separate 5000 gallon storage tanks. The results of measurements on the brine in each of these tanks are tabulated in Tables 6 and 7. The carbon used for treatment of this brine consisted of 840 pounds of partially spent and 180 pounds of fresh carbon. The value of the chemical oxygen demand dropped to about half of the influent value for the first 1000 gallons of effluent and then rose rapidly to a level approaching the influent level. The color transmittance reading did not start to decrease until after approximately 6000 gallons of effluent had been collected. These results suggest that the color of the effluent brine is a more sensitive index of extent of treatment than is the chemical oxygen demand.

Visalia Processing Waters

Lye rinse water - A lye rinse of approximately 1000 gallons was used on the carbon bed to remove organic acids. On February 10, a 180 pound portion of fresh carbon was placed on top of the column before the actual lye rinse water was pumped through the bed.

The lye rinse water analytical results are tabulated in Table 8. The suspended solids and COD were reduced considerably by the carbon bed. A reduction of 91 percent for the former and 62 percent for the removal of the latter was obtained. The caustic concentration became stabilized and very little was adsorbed by the carbon bed.

Transport brine - A fresh water counter flow portion of approximately 720 gallons was used to dilute the lye concentration within the carbon bed on March 10, 1969. Before initial pumping operations began, a charge of 120 pounds of fresh activated carbon was placed within the carbon tower. The holding tank was filled with spent transport brine and the pump operated at a flow rate of 2 gallons/min. The flow rate and screen were checked at intervals to maintain desired operating conditions, but the rate had to be adjusted constantly because of the clogging of the Dutch Corduroy wire cloth. The screen was replaced

TABLE 6

ANALYTICAL RESULTS ON RECONDITIONED VISALIA STORAGE BRINE -- 1

Cumulative Gallons	pH	Total Acidity % Lactic Acid	Color	Percent Transmittance at 425 mμ	NaCl, percent	COD, mg/liter	Suspended Solids ppm
0	4.0	0.42	Dk. Brown	33.0	1.65	22,300	64
834	5.6	0.07	Clear	100	1.45	10,650	98
1,670	4.2	0.41	Lt. Yellow	100	1.45	20,500	36
2,500	4.1	0.42	Lt. Yellow	100	1.60	21,900	51
3,340	4.0	0.42	Lt. Yellow	100	1.60	21,900	51
4,170	4.0	0.42	Lt. Yellow	100	1.60	22,100	28
5,000	4.0	0.42	Lt. Yellow	100	1.65	22,100	30

TABLE 7

ANALYTICAL RESULTS ON RECONDITIONED VISALIA STORAGE BRINE--2

Cumulative Gallons	pH	Total Acidity %Lactic Acid	Color	Percent Transmittance at 425 mμ	NaCl, percent	COD, mg/liter	Suspended Solids ppm
0	4.1	0.39	Dk. Brown	27	2.15	25,200	49
834	4.0	0.38	Lt. Yellow	100	1.95	23,750	25
1,670	4.1	0.38	Lt. Yellow	90	2.15	22,300	34
2,500	4.1	0.38	Yellow	84	2.15	21,100	13
3,340	4.1	0.38	Yellow	73	2.15	21,700	33
4,170	4.1	0.39	Yellow	78	2.15	21,500	56
5,000	4.1	0.39	Yellow	88	2.20	21,900	79

TABLE 8

CARBON COLUMN TREATMENT OF STRONG LYE RINSE WATER

	Light Transmittance at 425 mμ	Suspended Solids (ppm)	pH	%NaOH	%NaCl	COD mg/liter	COD Removal
Influent	62.0*	138	12.5	0.83	0.45	4432	----
Effluent Gallons							
75	96.0	0	9.5	0.03	-	492	3940
150	82.0	0	11.1	0.04	-	1379	3053
225	62.0	1	12.0	0.19	0.10	1379	3053
300	62.5	7	12.4	0.69	0.23	1379	3053
375	49.0	3	12.4	0.70	0.21	1674	2758
450	51.5	12	12.4	0.71	0.13	1970	2452
525	66.5	18	12.4	0.80	0.24	1477	2955
600	57.0	15	12.4	0.80	0.32	2167	2265
675	48.0	12	12.5	0.80	0.17	1773	2659
750	41.0	22	12.5	0.80	0.22	1871	2561
825	27.0	26	12.5	0.80	0.20	2167	2265
900	18.5	28	12.5	0.80	0.15	2167	2265

*Diluted with 4 parts of Distilled Water

with a vinyl cloth which had a much larger mesh opening. Olive oil coated the screen and created a partial vacuum against the pump and thereby decreased the flow rate.

Analytical results of the transport brine operation are tabulated in Table 9. The elution of sodium hydroxide is considered the most significant observation in the data. The salt content finally reached equilibrium at approximately 440 gallons. The COD removal was not as extensive as the adsorption of colored components and filtration of suspended particles.

Imported Spanish Olive Brine - On March 19, 1969, four plastic lined bins containing a total of 600 gallons of spent imported Spanish olive brine was recycled through the carbon tower at a flow rate of 2 gallons/min. The brine contained considerable quantities of top mold and suspended solids, so a filtering device was constructed using a siphon hose and a 24 x 110 Dutch Corduroy wire screen.

The analytical results of this operation are tabulated in Table 10. At first glance, the light transmittance reading indicates a rapid darkening of the brine with a corresponding decrease in pH. The salt content is much higher than in the domestic solutions. Suspended solids and chemical oxygen demand average removal was 30.3 percent for the former and 64.5 percent for the latter. The total acidity reduction was due to the elution of lye from the carbon bed.

Domestic Spanish Olive Brine - Immediately following the imported Spanish olive brine, the domestic variety was pumped through the carbon bed. Only two bins of domestic Spanish olive brine was obtainable at the time for our pilot plant studies. The same conditions prevailed in the pumping operations, but due to a small amount of olive oil, the screen used was unsuitable for that part of the operation and so a large vinyl screen was substituted.

A summary of the analytical results are tabulated in Table 11. The total acidity reduction was due to the lye elution from the carbon bed, but the final solution was immediately suitable as a neutral canning brine. The suspended solids were removed by 21.4 percent. This figure was arrived at by including the solids from the imported variety. The COD figure was calculated in a similar way; approximately 37.8% COD was removed.

TABLE 9

CARBON COLUMN TREATMENT OF TRANSPORT BRINE

	Light Transmittance at 425 mμ	Suspended Solids (ppm)	pH	%NaOH	% NaCl	COD mg/liter	COD Removal
Influent	54.0	200	7.2	0	3.10	744	744
Effluent Gallons							
80	100	0	10.3	0.044	0.02	495	249
200	100	0	10.0	0.007	2.20	0	0
320	100	0	9.7	0.007	2.27	0	0
440	100	0	10.1	0.011	3.05	495	249

TABLE 10

CARBON COLUMN TREATMENT OF IMPORTED SPANISH OLIVE BRINE

Influent Tanks	Light Transmittance 425 mμ	pH	%NaCl	Total Acidity	Suspend. Solids	COD mg/liter	COD Removal
1	41.0*	3.6	8.4	8.70	338	22,250	----
2	49.0*	3.5	8.4	6.46	204	15,280	----
3	47.0*	3.5	8.3	9.55	168	20,150	----
4	42.5*	3.5	9.7	8.44	226	18,850	----
Average	46.4	3.5	8.7	8.29	234	19,133	----
Effluent Gallons							
90	100	9.1	2.25	0	22	1,950	17,183
210	70	8.7	8.25	0	98	6,000	13,133
330	65	8.4	8.50	0	63	7,800	11,333
450	63	8.0	8.90	0.07	53	8,780	10,453
510	57	7.9	9.50	0.07	100	8,050	11,083
570	54	7.7	9.70	0.14	89	8,370	10,763

*Diluted with 4 parts of Distilled Water

TABLE 11

CARBON COLUMN TREATMENT OF DOMESTIC SPANISH OLIVE BRINE

	Light Transmittance 425 mμ	pH	%NaCl	Total Acidity	Suspend. Solids ppm	COD mg/liter	COD Removal
Influent	34	3.25	6.0	3.10	264	5,690	--
Effluent Gallons							
60	63	7.5	8.8	0.14	62	7,460	4,951
120	65	7.5	6.85	0.14	60	7,300	5,111
150	65	7.5	6.70	0.14	38	8,370	4,041

EVALUATION OF THE REUSE POTENTIAL OF CARBON TREATED BRINES

Storage of Olives

A critical test in this demonstration project was the storage of freshly harvested olives in carbon column reconditioned brine on a commercial scale. The majority of the plants in the California Olive industry use strong brines (6 to 10 percent sodium chloride) to store a large fraction of each year's harvest. If the used brine could be reconditioned by carbon treatment and reused each year for the storage of freshly harvested olives, the need for disposal of a large quantity of salt would be avoided. This reuse rather than discharge of strong salt brines would reduce the danger of saline pollution of receiving waters.

Therefore, the experimental storage of fifteen tons of olives in 1530 gallons of reconditioned brine was followed with intense interest. The olives used were medium Manzanillos and the brine had a starting salometer reading of 24° at the time storage started on October 23, 1968; the salt content of the brine was increased to 30° during the first two weeks of storage by the addition of solid sodium chloride. After three months of storage, the salt concentration was raised to 10% (40° salometry). A sample of olives was taken from the storage tank each week and examined for signs of spoilage or shrivel. The olives appeared to be undergoing a normal fermentation and storage behavior. Small portions of the stored olives were processed and canned starting on December 23, 1968 and February 24, 1969. An additional sample was canned during the week of April 21, 1969. A control sample of olives from normal storage tanks was obtained for each of the canning periods of the experimental olives.

Taste panel comparison of the olives stored in reconditioned brine and the olives stored in virgin brine will be made during the next report period of this demonstration grant.

Storage of Strong Brine

To get the most efficient use of a commercial scale carbon column unit it would be desirable to operate it several months before

freshly harvested olives are received for storage. For this type of operation, it would be necessary to store reconditioned brine in the absence of olives for periods of three to six months. It has been demonstrated on many occasions that used olive storage brines develop yeast, mold and bacterial growth in the absence of green olives. These changes in the storage brine make it unsuitable for reuse because olives stored in such brines produce a final product of inferior flavor and texture.

There is still considerable question about the storage performance of carbon treated brine in the absence of green olives. For this reason, a 5450 gallon portion of the 24° salometer brine from the brine reconditioning unit was stored in a redwood tank starting on October 24, 1968. The pH of the brine was adjusted from 4.1 to 3.7 by the addition of 20 gallons of 37% (food grade) hydrochloric acid. The brine in this storage tank developed a yeast or mold pellicle after 3 to 4 weeks of storage. The brine in the tank was reported by plant personnel to have an undesirable odor after three months of storage; however, project personnel could detect no significant undesirable odor on smelling the brine after three and four months of storage.

Storage of Weak, Acidified Brines

The 10,000 gallons of storage brines treated by the carbon column at Visalia were stored in two 5000 gallon tanks. The reconditioned brine had an average salt content of 1.8 percent, a pH of 4.0 and a total acidity of 0.29 percent. These storage brines were adjusted to lower pH's by the addition of a mixture of lactic and acetic acids. The brine in the two tanks was circulated by pumping to break up surface growth of yeasts and molds. The brine had a good odor and appearance after six weeks of storage in the absence of green olives.

Reuse of Processing Water

The recycled weak lye processing waters of Lindsay were transfered to Consolidated Olive Growers for their evaluation on the potential reuse of this solution in their lye curing operations. The plant personnel evaluated the potential reuse of this lye water and found no change in appearance or flavor.

Before it went through the carbon, the water was dark brown and had a pH of 9.5. After passing through the carbon the color was light yellow and the pH was 12.0. Evidently some lye previously run through the carbon got into this water. This re-claimed water was left on the olives all night following the second lye. Then the water was thrown away. The flavor of the olives after process seemed to be the same as olives having normal fresh water changes.

Reuse of Lye Rinse Water

The recycled strong lye rinse water was used by the Visalia plant as make up water in their lye tanks. The plant personnel evaluated the potential reuse of this type of lye rinse water and found this to be satisfactory.

Another potential reuse of recycled lye rinse water is in neutralizing the organic acids in the regenerated brines and thereby using this mix as a canning brine, with the proper salt content and pH adjustment. This would reduce the pollution effect of chlorides discharged to the municipal sewer systems. An actual mix was made at Visalia and used as a canning brine.

Use of Reconditioned Brines as Canning Brines

On February 16th a canning brine was made from the reconditioned brine and lye water tanks at Visalia. Table 12 summarizes the results of this blending operation.

TABLE 12

PREPARATION OF CANNING BRINE FROM CARBON COLUMN TREATED BRINES

Batch	Tanks	Vol 50-50 Mix	pH	NaCl	ml 11% NaOH Needed	NaCl Needed to 13.5°S
A	693 - 694	18 qt.	4.2	6.5°S	123	46 oz.
B	693 - 692	18 qt.	4.2	7.0°S	127	46.5 oz.

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Corvallis, Oregon 97330

As the 50-50 mix was neutralized a color change from light yellow to deep purple developed. As the final mix was heated to 190°F for the canning operation a precipitate formed which in turn settled to the bottom of the container. The final solution was light gray in color.

The hot liquid was used as a canning brine and twelve 300 cans of batches A and B were obtained with their control counterpart.

On February 27, 18 quarts each of 692 and 694 tanks at Visalia were sampled and canned. The liquid canning brine did not produce a color change or a precipitate, as with the neutralized brine and had a very good fruit odor. Twelve 300 cans of each tank were obtained with their controls.

On March 10, twelve 300 cans of Manzanillo olives, with recycled transport brine as canning brine, were packed by the Visalia plant and control samples obtained for comparison.

On March 20, 12 cans of 13°S olive brine produced from 46°S recycled, imported Spanish olive brine was used as canning brine on ripe olives. Twelve glass jars of imported Spanish Olives were filled with 21°S brine obtained from the recycled imported Spanish Olive brine. Control samples were obtained whenever possible.

The canned olives are stored at the Berkeley laboratory and are to be used in taste panels in evaluating the effects of using these different brines.

Reuse of Spanish Olive Brines

The recycled Spanish Olive brines can be used as a filling brine. Spanish Olives are normally stored in wooden barrels and the liquid level therein must be readjusted periodically with fresh salt brine. Additional salt would not be needed for this purpose.

A canning brine could easily be produced by dilution with water and neutralizing the acids, if any, with sodium hydroxide solution.

Another potential use of this high salt content brine, is in the make up water production as storage brines for freshly harvested olives.

LABORATORY SCALE BRINE TREATMENT UNIT

Description of Bench Top Scale Unit

The exceptionally large olive crop harvested in 1968 made it difficult for the olive companies to provide storage tanks for the experiments originally planned for the project. The frequent rains in the winter of 1968-1969 made field operation of the brine reconditioning unit very difficult. For these reasons, a bench top scale carbon column unit was assembled in the laboratory for preliminary studies on dilute olive processing brine.

A schematic drawing of the unit is shown in Figure 7. A photograph of the unit is illustrated in Figure 8. The major components of the unit are pigmented polyvinyl chloride tubes two inches in diameter and five feet in length. The brine was delivered to the tubes by a small metering pump. A tubing arrangement allowed passage of the brine through four tubes in series or through each tube separately. Each tube was packed with 1600 g (3.53 pounds) of CAL type 12 x 40 activated carbon.

Operation of Bench Top Unit

The initial use of the bench top unit was in the treatment of a sample of vat room water from Lindsay. This water represents a large volume, dilute brine from the lye curing, rinsing and aeration operations during the production of black ripe canned olives. The disposal of these processing waters is a difficult problem. If the waters could be reclaimed by carbon treatment for at least one reuse cycle, the disposal problem would be significantly reduced.

A 40 gallon sample of Lindsay processing water was put through the bench top unit. At the start (for the first 23 liters) the brine was pumped through all four carbon filled tubes in series. The low flow rate of the unit when all four tubes were used in series made it necessary to pump the remaining 129 liters through one tube at a time.

Results from the Use of the Bench Top Unit

The influent Lindsay processing water had the following partial composition: chemical oxygen demand, 6,400 mg/liter; suspended

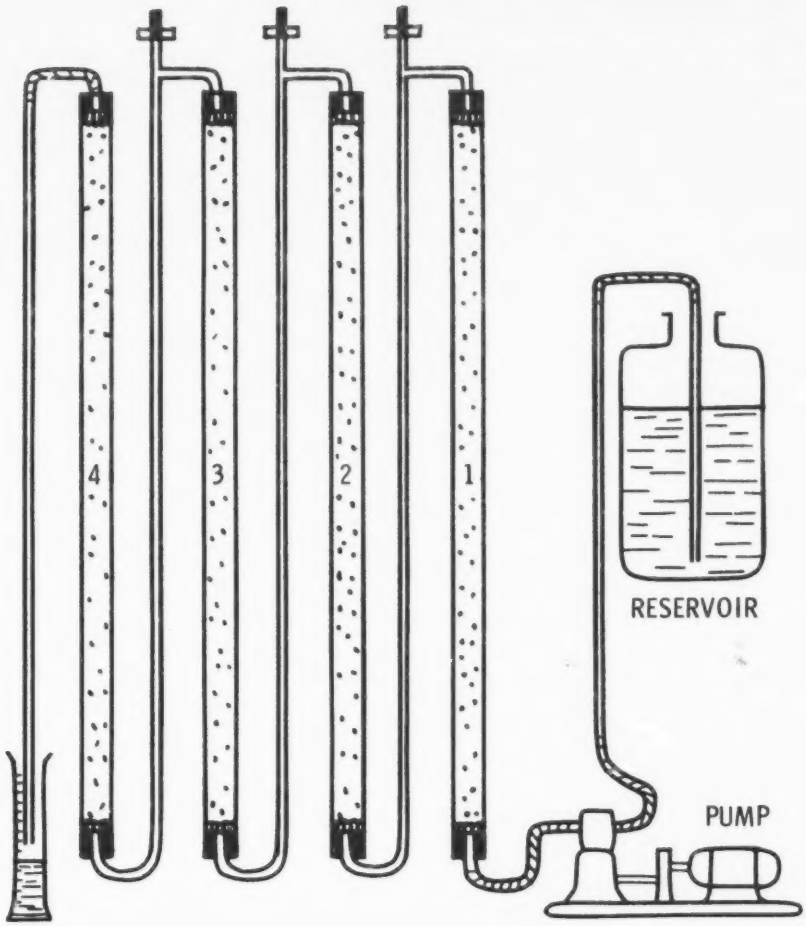

Figure 7. Laboratory Bench Top Carbon Column Assembly - Schematic

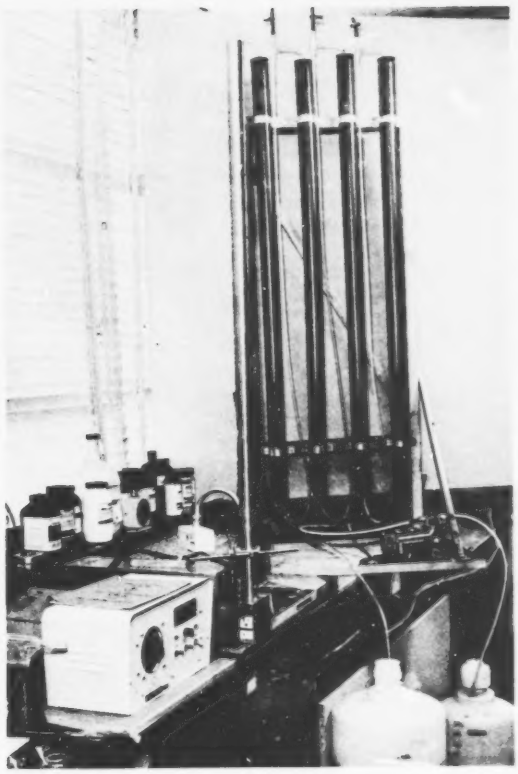

Figure 8. Laboratory Bench Top Column- Photograph

solids, 68 ppm; pH, 11.2; sodium chloride content 0.2 percent (2,000 ppm). The light transmittance of the influent brine, after dilution with 5 parts of distilled water, was 78 percent. The results of previous experiments had demonstrated that the salt content of brines was not changed by carbon treatment, so no further analyses for salt content were made. Table 13 tabulates the results found for changes in processing water after carbon treatment.

The results obtained with the bench top scale unit were sufficiently encouraging (especially for pH reduction, color removal and COD reduction) that larger volumes of processing waters were put through the large unit and their reuse potential conducted as described above in this report.

TABLE 13

RESULTS OF TREATMENT OF LINDSAY PROCESSING WATER IN LABORATORY BENCH TOP CARBON COLUMN UNIT

Volume Treated, liters	Flow Rate ml/min.	Percent Transmittance at 425 millimicrons	COD Removal mg/liter	Suspended Solids ppm	pH
0	0	78.0 (1+5 dilution)	--*	68	11.2
1	2.46	100	"	--	8.8
2	"	"	"	--	9.1
3	"	"	"	--	8.5
4	"	"	"	--	8.4
5	"	"	"	--	8.4
6	"	"	"	--	8.5
7	"	"	"	--	8.4
8	"	"	"	--	8.5
8.9	"	"	"	--	8.6
9.1	"	"	"	--	8.7
9.2	"	"	"	--	8.9
9.3	"	"	"	--	9.2
10.0	"	"	"	--	9.2
11	"	"	"	--	9.4
12	"	"	"	--	9.3
13	"	"	--	--	9.1
14	"	"	--	--	9.2
15	"	"	--	--	9.3
16	"	"	--	--	9.6

TABLE 13. (Con't.)

Volume Treated, liters	Flow Rate ml/min.	Percent Transmittance at 425 millimicrons	COD Removal mg/liter	Suspended Solids ppm	pH
17	2.46	100	--	--	9.3
18	"	"	--	--	9.3
19	"	"	--	--	9.7
20	"	"	--	--	9.6
21	"	"	--	--	9.4
23	"	"	--	--	9.3
24	"	"	3744	4	10.1
25	35.0	"	--	--	10.1
26	"	"	--	--	10.1
27	"	"	3744	1	10.0
28	"	"	--	--	10.1
29	"	"	--	--	10.1
30	"	"	--	--	10.1
31	"	"	3648	8	10.2
32	"	"	--	--	10.2
33	"	"	--	--	10.3
34	"	"	--	--	10.3
35	"	"	3552	2	10.3
36	40.0	"	3936	5	10.4
37	"	"	--	--	10.3
38	"	"	--	--	10.3
39	"	"	3072	10	10.3
40	"	"	--	--	10.2
41	"	"	--	--	10.2

TABLE 13. (Con't.)

Volume Treated, liters	Flow Rate ml/min.	Percent Transmittance at 425 millimicrons	COD Removal mg/liter	Suspended Solids ppm	pH
42	45.0	100	3360	3	10.0
43	"	"	--	--	9.8
44	"	"	--	--	9.6
45	"	"	--	--	9.5
46	"	"	2976	8	9.5
47	"	"	--	--	"
48	"	"	--	--	"
49	"	"	3360	9	"
50	"	"	--	--	"
51	"	"	--	--	"
52	"	"	3264	10	"
53	"	"	--	--	"
54	"	"	--	--	"
55	"	"	3168	11	"
56	"	"	--	--	"
57	"	"	--	--	"
58	"	"	3072	13	9.6
59	"	"	--	--	"
60	46	"	--	--	"
61	"	"	2697	14	8.6
62	"	"	--	--	8.3
63	"	"	--	--	8.4
64	"	"	2976	13	8.4
65	"	"	--	--	8.5

TABLE 13. (Con't.)

Volume Treated, liters	Flow Rate ml/min.	Percent Transmittance at 425 millimicrons	COD Removal mg/liter	Suspended Solids ppm	pH
66	46	100	--	--	8.5
67	"	"	2976	14	"
68	"	"	--	--	"
69	"	"	--	--	8.6
70	"	"	3069	16	8.7
71	"	"	--	--	"
72	"	"	--	--	"
73	"	"	2976	18	8.8
74	"	"	--	--	8.8
75	"	"	2883	20	8.9
76	"	"	--	--	9.0
77	"	"	--	--	9.0
78	"	"	2883	20	9.0
79	"	"	--	--	9.1
80	"	"	2883	--	"
81	"	"	--	--	"
82	"	"	--	--	"
83	39	"	1248	23	7.4
84	"	"	--	--	7.6
85	"	"	--	--	7.7
86	"	"	--	--	"
87	"	"	2016	15	"
88	"	"	--	--	"
89	"	"	2400	20	"

TABLE 13. (Con't.)

Volume Treated, liters	Flow Rate ml/min.	Percent Transmittance at 425 millimicrons	COD Removal mg/liter	Suspended Solids ppm	pH
90	42	100	--	--	7.7
91	"	"	1300	19	7.3
92	"	"	--	--	7.3
93	"	"	--	--	7.5
94	"	"	1600	25	7.5
95	"	"	--	--	7.5
96	"	"	--	--	7.3
97	"	"	1900	21	7.5
98	"	"	--	--	7.4
99	"	"	--	--	7.4
100	"	"	2000	27	7.5
101	"	"	--	--	"
102	"	"	--	--	"
103	"	"	1900	20	"
104	"	"	--	--	"
105	"	"	--	--	"
106	"	"	1100	19	7.6
107	"	"	--	--	7.6
108	"	"	--	--	7.6
109	46	85	--	--	7.3
111	"	76	--	--	7.2
113	"	85	--	--	"
115	"	"	--	25	"
117	"	84	--	--	"

TABLE 13. (Con't.)

Volume Treated, liters	Flow Rate ml/min.	Percent Transmittance at 425 millimicrons	COD Removal mg/liter	Suspended Solids ppm	pH
119	46	84	--	--	7.2
121	"	79	--	36	7.3
123	"	81	--	--	7.3
125	"	79	--	--	7.4
127	"	79	--	41	7.5
129	"	83	--	--	7.4
130	"	85	--	--	7.4
131	62	80	2860	83	7.5
133	"	61	--	--	7.5
135	"	58	--	--	7.6
137	"	56	--	--	7.6
139	"	56	3375	31	7.5
141	"	53	--	--	7.5
143	"	53	--	--	7.5
145	"	53	3170	44	7.6
147	"	50	--	--	"
149	"	50	--	--	"
151.5	"	44	3170	46	"

* No measurements made

GENERAL METHODOLOGY

Volume Measurements

A simple measurement, to obtain the flow rate volume from the carbon tower, was employed using a stop watch and pre-calibrated plastic container. Tank volumes were either calculated from dimensions or from flow rate data.

Sampling

Grab samples from the influent, effluent and tower port spigots were obtained at specified intervals of time. Quart glass containers were used whenever possible to eliminate contamination and spillage of sample. Each container was clearly labeled by sampling point, pump rate, date, and time of sampling.

Sample Storage

Samples from the carbon tower were immediately taken to the plant laboratory refrigerator for temporary storage. After completion of a regular experiment the samples were gathered and transported in polystyrene ice chests for the return trip to the Berkeley laboratory. At the destination the samples were refrigerated for subsequent analysis.

Methods of Analysis

The following analytical procedures were employed on samples when appropriately applied to a given sequence of samples.

Chemical Oxygen Demand - The Jeris Modified COD tests (4) was employed on all analytical data presented in the report. This "rapid" COD procedure involves the addition of waste sample to a flask containing mercuric sulfate. After mixing, 25 ml of dichromate-acid - silver sulfate mixture is added, mixed, and placed on a pre-heated hot plate. A thermometer is inserted in the flask, and the temperature is checked as the mixture is heated. When 165°C is reached, the sample is withdrawn and cooled. (Care must be employed in heating the sample because of the rapid rise in temperature.)

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Water is added cautiously, and after further cooling, the solution is titrated with ferrous ammonium sulfate in the presence of ferroin indicator.

(Chemical reagents)

a. Dichromate - acid solution 0.05N. Dissolve 5 g $K_2Cr_2O_7$ and 20 g $AgSO_4$ in a solution consisting of 1 L each of concentrated H_2SO_4 and H_3PO_4.

b. Ferrous ammonium sulfate, 0.05N. Dissolve 19.5 g of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ in distilled water, add 20 ml of concentrated H_2SO_4 and dilute to 1 L. This solution is standardized against O.05N $K_2Cr_2O_7$ prior to use.

c. Ferroin indicator solution. Dissolve 1.485 g 1,10 phenanthroline monohydrate and 0.695 g $FeSO_4 \cdot 7H_2O$ in 100 ml distilled water.

d. Mercuric sulfate, A. R. crystals.

e. Dichromate standard, 0.05N. Dissolve 2.5418 g $K_2Cr_2O_7$ dried, primary standard grade, in distilled water and dilute to 1 L.

All of the above are ACS reagent chemicals unless otherwise noted.

(Procedure)

a. Add approximately 0.3 g of $HgSO_4$ directly to a 500 ml Erlenmeyer flask (with a little practice, this may be measured from the tip of a spatula without weighing, in fact an excess may be helpful to compensate for the NaCl.)

b. Pipet 5 ml from an aliquot (5/100 ml) of the diluted sample into the flask for a brine solution and 5 ml from an aliquot (10/100 ml) into the flask for lye rinse water. Mix well.

c. Two blank are run by substituting 5 ml of distilled water for the sample and continuing with steps "d" to "h". Use the average of two titrations in the calculations.

d. Carefully add 25 ml of dichromate-acid solution to the flask and swirl the contents.

e. Handling the flask by the neck with tongs, place the Erlenmeyer flask on a pre-heated hot plate and heat the solution to 165 ± 1°C. A thermometer should be immersed in the tilted flask to read the temperature correctly.

f. Cautiously add approximately 150 ml of distilled water to the partially cooled mixture. (The addition of water to a very hot

acid mix does induce vigorous boiling and spattering.)

g. Cool the solution in a water bath to ambient temperature.

h. Add 3 drops of ferroin indicator and titrate with ferrous ammonium sulfate solution from a blue green to a red-brown end point.

(Calculations)

The COD is calculated as follows:

$$N = \frac{(25)(0.05)}{\text{ml from Step "i"}} \qquad \text{Eg. 1.}$$

Where N = normality of the Ferrous ammonium sulfate solution.

Eg. 2

$$\text{COD (mg/l)} = \frac{N \times 8000 \times (\text{ml from Step "c"} - \text{ml from step "h"})}{\text{ml of Sample}}$$

Suspended Solids - A millipore filter apparatus with a 47 m. m., Type E, glass fiber filter disc is employed in this procedure. The filter disc is dried in an oven at 103°C for 1 hr. (30 min. in a mechanical convection oven), cooled to room temperature in a desiccator and weighed. The filter disc is placed in the suction apparatus with tweezers. A volume of sample to be taken for filtration will depend on the concentration of the suspended matter in the sample and should be as large as practical. 100 ml portions are used throughout the report unless otherwise noted. A well mixed sample is measured out with a graduated cylinder and filtered slowly by suction.

Leaving the suction on, wash with 10 ml distilled water to remove soluble salts. Dry the filter disc and solids at 103°C for 1 hour as previous and allow the disc to cool to room temperature in a desiccator before weighing.

The suspended solids are calculated in ppm

$$\text{ppm S. S.} = \frac{R \times 10^6}{\text{Sample volume}}$$

where R = weight of dried residue.

Color - The color of the waste samples is measured in a Bausch & Lomb model 20 Spectrophotometer. The instrument is standardized at 100% transmittance with distilled water at the desired

wave lengths of 425 and 450 mμ. Samples are read directly in % transmittance and readings converted to optical density. Aliquots are necessary if readings fall below 10% transmittance.

pH - The measurement of pH of a solution is obtained by the use of a commercial electrometric portable pH meter. The meter is first standardized with a buffered pH solution. The appropriate probes are immersed into the solution under test and the pH determined by reading the value directly from the meter.

Total Acidity - In a food plant material, total acidity is a measure of the amount of acidic substances principally present as organic acids and carbon dioxide. A 10 ml sample is placed in a 150 ml beaker. Add approximately 40 ml of distilled water. Using a glass electrode pH meter, titrate with 0.1560 N Sodium hydroxide with constant stirring to an end point of pH 8.3. Make sure the tip of the buret is clean and free from solid alkali. Read the number of ml of sodium hydroxide used to one-tenth of a ml, and divide by ten. A correction factor of 1.405 is used to convert the results into percent of acid in terms of lactic acid.

Salt Content - Pipet 5 mls of an aliquot (5/100 for Brine) into a 100 ml beaker. Add approximately 25 ml of water. Use a PHOTOVOLT Model 115 Electronic pH meter with the silver and calomel electrodes. Immerse the electrodes in the solution and set the main switch to reference. Adjust the pH scale at 7.0 with the standardizing control knob. Titrate with .0855 N silver nitrate (do not titrate with silver nitrate solution that has been exposed to light in the buret for more than a few hours), with constant stirring until the needle rests at 11.5. Read the number of ml of silver nitrate used to one-tenth of a ml and divide by ten. The result is the percent salt.

Percent Sodium Hydroxide - A suitable sample of lye water (less than 25 ml) is titrated with 0.0908 N H_2SO_4 with stirring to an end point of pH 7.0. The percentage of NaOH in the lye water is given by:

$$\% \text{ NaOH} = \frac{4 (A \times B)}{C}$$

Where: A = volume of acid used
B = normality of the acid
C = size of sample to be titrated, in ml

APPENDIX A

Project Title:	Reconditioning and Reuse of Food Processing Brines
Project Numbers:	WPRD - 134 - 01 - 68
	RF 5630 (Berkeley)
Sponsoring Organizations:	Federal Water Pollution Control Administration U. S. Department of the Interior Olive Advisory Board Olive Administrative Committee
Project Officer:	Mr. William H. Pierce, Sanitary Engineer California/Nevada Basins Southwest Region Federal Water Pollution Control Administration 620 Central Avenue, Building 26 Alameda, California 94501

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